National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2030a

Glass Filter for Transmittance Measurement

This Standard Reference Material (SRM) is a primary transfer standard certified using a national reference spectrophotmeter at NIST [1-3]. It is intended for use in the one-point verification of the transmittance and absorbance scales of spectrophotometers at the given wavelength and measured transmittance. It consists of one glass filter in its holder and one empty filter holder. The exposed surface of the glass is approximately 29 mm x 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see Figure 1). The filter bears an identification number. For protection, the metal holder is provided with two shutters that should be removed during measurements.

Certified Values of Transmittance Density and Transmittance: The certified transmittance density value independently determined for the filter at 22 °C ± 1 °C and at a wavelength of 465.0 nm is given in Table 1. This value is calculated from measured transmittance (T) as $-\log_{10}T$ and should be indicated by the absorbance (A) scale of the spectrophotometer when measured relative to air. The corresponding certified transmittance value is also given in Table 1. The expanded uncertainties for the certified transmittance density value in Table 1 are calculated from uncertainty components given in Table 2 (see Determination of Expanded Uncertainties). The expanded uncertainty for the transmittance value is calculated from the transmittance density uncertainty. The expanded uncertainty allows for possible changes due to slight surface contamination and fundamental materials effects over the two-year period of certification. The maximum value of spectral bandpass for which the certified value is valid is 2.7 nm.

Expiration of Certification: This certification is valid within the measurement uncertainties specified for two years from the date of certification given for this filter in Table 1, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is altered, contaminated, or damaged. The filter may be returned to NIST for cleaning and recertification at two-year intervals. Recertification can be arranged by contacting the NIST Optical Filters Program at (301) 975-4115.

The overall direction and coordination of technical measurements leading to certification were performed by J.C. Travis and G.W. Kramer of the NIST Analytical Chemistry Division.

Transmittance measurements were performed by M.V. Smith of the NIST Analytical Chemistry Division.

Statistical support was provided by K.R. Eberhardt and H-k Liu of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.W.L. Thomas.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 10 August 2000
See Certificate Revision History on Page 4

Nancy M. Trahey, Chief
Standard Reference Materials Program

Instructions for Use: The transmittance of the filter depends upon the intrinsic properties of the material and the wavelength, spectral bandpass, and geometry of the optical beam. It can be affected by other factors such as stray light, temperature, and positioning of the filter. Changes in the transmittance may be caused by changes in surface conditions, aging of the glass, exposure to a harmful atmosphere, or careless handling as indicated (see Storage and Handling) [1,4-6]. The measurement wavelength of 465.0 nm should not be in error by more than 1 nm, and the maximum spectral bandpass value of 2.7 nm should not be exceeded. The wavelength axis of the instrument may be calibrated using NIST SRM 2034 Holmium Oxide Solution Wavelength Standard.

Instrument verification should be performed at an ambient temperature between 20 °C and 24 °C [4]. The empty filter holder provided is to be used in the reference beam of the spectrophotometer so that approximately equivalent conditions of stray radiation are achieved for both beams. The shutters provided with the filter must be removed at the time of measurement and be replaced after the measurements have been completed. Measurements performed outside of these specified conditions or the optical geometry used for certification (see Determination of Transmittances) could produce transmittance values that differ from the certified values.

To demonstrate that a user's measurements are traceable within acceptable limits to the accuracy defined by SRM 2030a, the user must first determine the required tolerances or acceptable uncertainty for the application in question. It is recommended that a number of replicate measurements be made with removal and replacement of the filter between replicate measurements. The user should then compare the mean value and the user-defined tolerance with the NIST-certified value and expanded uncertainty (Table 1). An acceptable level of agreement between a user's measurement and the certified value is assured if any part of the range defined by the NIST certified value and its expanded uncertainty overlaps any part of the user's tolerance band defined by the measured mean and the user-defined level of acceptable uncertainty [7].

Storage and Handling: SRM 2030a is stored in a black anodized aluminum container to minimize contamination of the glass surface with particulate matter due to static charge. The filter is placed in a cylindrical cavity to prevent any contact between the filter face and the walls of the storage container. The filter holder is provided with a flat leaf spring that is inserted into the cylindrical cavity to minimize damage during transportation. This spring should be removed during normal use. **Improper storage or handling of the filter may cause changes in the transmittance** [4]. It is recommended that the filter in the holder be handled only by the edges with soft, powder-free, plastic (polyethylene) gloves and optical lens tissue. When not in use it should be stored in the container. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided. If the surface of the glass filter becomes contaminated, no attempt should be made to clean it. However, dust may be removed by using a rubber-bulb air puffer without contacting the surface of the filter. The SRM should be returned to NIST for recertification.

Instrument Dependence Warning: Instruments for which wavelength dispersion occurs after the light has passed through the filter are particularly susceptible to minor deviations in the optical beam by the SRM unit. If such effects are detected or suspected, the user should contact J.C. Travis, NIST Analytical Chemistry Division at (301) 975-4117, for assistance and instructions.

Source and Preparation of Material: The neutral glass for the filter was provided by either Starna Cells Inc., Atascadero, CA or Schott Glass Technologies, Inc. of Duryea, PA from samples of Schott NG-5 glass, selected for best homogeneity and minimal inclusions and striae[1]. The filter was ground and polished to the appropriate thickness to achieve the nominal transmittance of 0.3 [1,4,5]. SRM 2030a has been polished to a flatness of one wavelength of the helium-neon laser 633 nm line over the central 5 mm x 20 mm filter area and to a parallelism of 0.1 mrad or better. Prior to certification measurements, the glass filter was aged at NIST for at least six months, and each filter was examined for surface defects and thoroughly cleaned [4].

Determination of Transmittances: The transmittance measurements were made relative to air (an empty filter holder) at an ambient temperature of 22 °C ± 1 °C using a high accuracy spectrophotometer designed and built in the

[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

NIST Analytical Chemistry Division [3]. This instrument represents a primary transmittance standard; its transmittance accuracy is established using the double-aperture method of linearity testing [3,4,6,8]. The effective spectral bandpass used to determine the certified values was 0.8 nm. The transmittance measurements were made by projecting the vertical image of the slit (approximately 6 mm x 1 mm) onto the middle of the entrance face of the glass filter using 1:1 imaging and a convergent beam geometry with an aperture ratio of f/10. The filter was mounted in a multiple filter carriage in the spectrophotometer. The transmittance value reported in Table 1 is the average of three transmittance values over the several minute time period required for three carriage cycles. The filter was measured in the spectrophotometer in a position perpendicular to the incident light beam as shown in Figure 1. The transmittance value was calculated from a measurement of the intensity transmitted through the filter and bracketing measurements of the intensity transmitted through an empty filter holder with a settling time of approximately 5 seconds followed by a signal integrating time of approximately 2 seconds. Transmittance was determined in this way several times during an aging period of at least six months. Only the final measurement was used as the basis of the certified value.

Uniformity: The transmittance uniformity for SRM 2030a was tested at 546 nm (with a 30 nm effective spectral bandpass) using a commercial spectrophotometer to compare the transmittance density measured at the center of each filter with that measured 7 mm below the center. Filters were rejected if the relative difference of the two readings exceeded the allowable limit of 0.0013 absorbance units with a statistical confidence of 95 %. This limit is reflected in Table 2 and was established experimentally for prior filters of this type by sampling 1 mm x 4 mm areas over a region 5 mm wide by 20 mm long and located symmetrically about the center face of each filter [9].

Determination of Overall Uncertainties: The expanded uncertainties (U) of the certified transmittance density values of Table 1 are determined from combined standard uncertainties (i.e., estimated standard deviations) of component uncertainties reported in Table 2 and a coverage factor $k = 2$ based on the Student's t-distribution for more than 30 degrees of freedom [10]. This uncertainty includes “Type A” uncertainties which are evaluated by statistical methods, and “Type B” uncertainties which are determined by other means. The standard uncertainties are combined by the root-sum-of-squares method. The expanded uncertainty defines an interval within which the unknown value of the transmittance density can be asserted to lie with a level of confidence of approximately 95 %.

The Type A standard uncertainty component was determined from the results of a statistical analysis of five measurements taken on each of two filters and on each of six days. The pooled standard deviation of replicates, s_p, was computed and reported as the standard uncertainty for 50 degrees of freedom.

The Type B uncertainty components of Table 2 were estimated from studies described in NIST Special Publication 260-116 [4]. The Type B uncertainty components are derived from an estimate of the range, $\pm\ a_i$, with the assumption that the uncertainty is uniformly distributed. The resulting standard uncertainty component is then approximated as $a_i \div \sqrt{3}$ [10].

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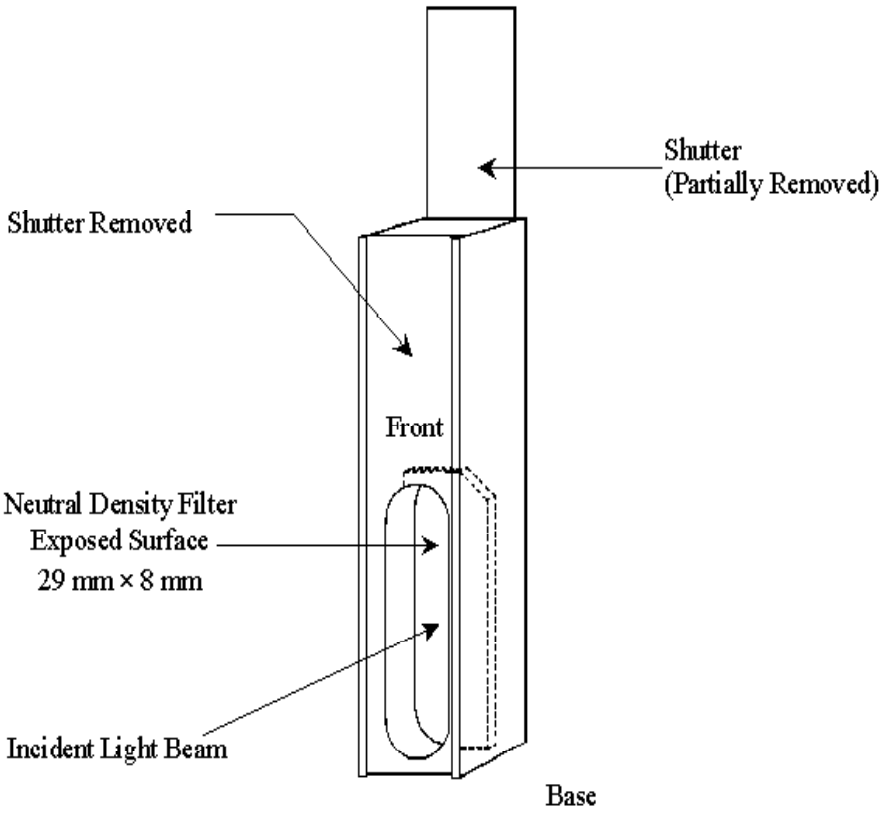

Figure 1. Metal Holder for the Colored Glass Filter

Certificate Revision History: 10 August 2000 (Technical changes due to new instrumentation.); 18 October 1993 (Editorial change); 11 August 1993 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.